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Materials Research Capabilities

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Materials Division

Lewis Research Center
Cleveland, Ohio

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NASA
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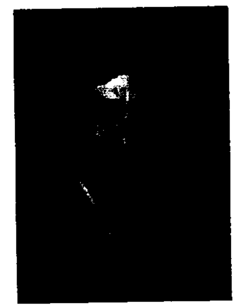


Foreword

Lewis Research Center, in partnership with U.S. industry and academia, has long been a major force in developing advanced aerospace propulsion and power systems. One key aspect that made many of these systems possible has been the availablity of high-performance, reliable, and long-life materials. To assure a continuing flow of new materials and processing concepts, basic understanding to guide such innovation, and technological support for development of major NASA systems, Lewis has supported a strong in-house materials research activity. Our researchers have discovered new alloys, polymers, metallic composites, ceramics, coatings, processing techniques, etc., which are now also in use by U.S. industry.

This brochure highlights selected past accomplishments of our materials research and technology staff. It also provides many examples of the facilities available with which we can conduct materials research. The nation is now beginning to consider integrating technology for high-performance supersonic/hypersonic aircraft, nuclear space power systems, a space station, and new research areas such as materials processing in space. As we proceed, I am confident that our materials research staff will continue to provide important contributions which will help our nation maintain a strong technology position in these areas of growing world competition.

Andrew J. Stofan
Director


NASA

The NASA Lewis Research Center is located directly west of Cleveland Hopkins International Airport in Cleveland, Ohio. This Center covers over 300 acres and includes a staff of 2600 professional and support personnel. Applied research in the areas of aeronautical, space and terrestrial propulsion, and power is done in partnership with industry, academia, and other government agencies.

Facilities at Lewis are among the finest in the world for developing and testing new materials, concepts, and processes. A variety of engineering test cells are available for testing components in simulated operating environments. Large facilities which can test complete systems in various environments include the supersonic wind tunnels, space simulation chambers, a 420-foot zero gravity facility, and altitude chambers for aircraft engines. A new microgravity materials science laboratory is available for visiting scientists and engineers to test potential shuttle and space station payload experiments.

This brochure highlights current materials research activities and describes some of the unique facilities which contribute to Lewis' materials research capabilities.

Materials Research Capabilities

Materials research at the Lewis Research Center focuses on NASA's need for long-range technology innovation, basic research in materials, and project support. Major thrusts are in the areas of advanced aerospace propulsion, advanced power systems, and microgravity science materials and applications.

Presently, the Materials Division staff consists of more than 130 chemists, metallurgists, ceramicists, polymer scientists, composite engineers, technicians, etc. Seventy percent of the staff hold advanced degrees, 45 percent of those being PhD's.

Current emphasis in metals research is directed toward single-crystal alloys, intermetallic compounds, refractory metals, and composite materials for use in engine components for the space shuttle, advanced hypersonic aircraft, and power systems for the space station. Polymer matrix composites are being investigated for potential use in aircraft engines and space structures. Ceramic research is seeking to better understand and control the microstructure-property relations in high-temperature structural ceramic systems. The microgravity environment of the shuttle and space station offers containerless ceramic processing as an alternative to current processing methods. Ceramic and metallic coatings are being developed to provide protection for engine components against attack in high-temperature, corrosive/erosive environments. Tribological experiments will yield a better understanding of the behavior of interfaces (e.g., solid-to-solid contact) in such mechanical systems as heat engines, aircraft components, and space mechanisms. All these research activities are supported by characterization laboratories which provide analysis and documentation of the chemical composition and microstructure of advanced materials, metals and nonmetals.

Patents, research reports, and nationally competitive technical awards provide a means to exchange knowledge and a way to recognize the efforts made in pursuing materials research at NASA Lewis.

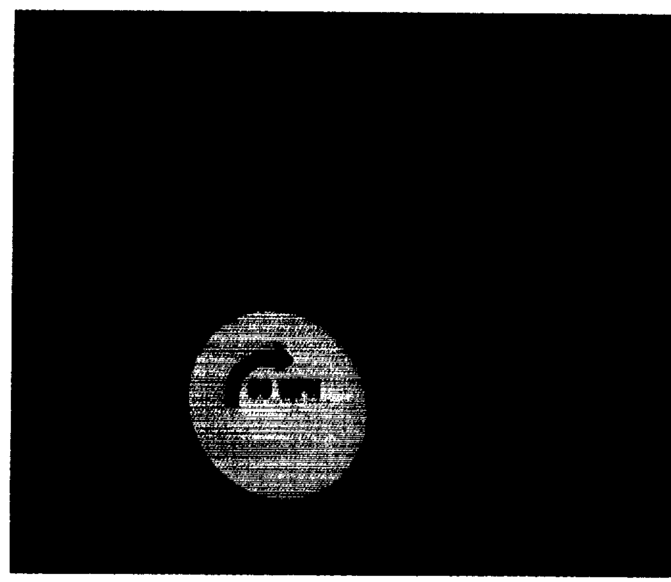
Advanced Metallic Materials

Research is underway on advanced metallic materials such as single-crystal alloys, copper alloys, intermetallic compounds, refractory metals, and composite materials. Studies cover such disciplines as deformation behavior, processing, fabrication, and joining; corrosion/compatibility; and mass transfer. Research efforts involving cooperative programs with other government agencies, private industry, and universities are aimed at gaining a fundamental understanding of the behavior of metallic materials under extreme environments.

Applications for these materials include advanced space power concepts, such as solar dynamic systems and nuclear powered systems required to provide electric power for the space station; turbine and nozzle components in the space shuttle main engine and orbital transfer vehicles; and engine components in advanced hypersonic aircraft.

Melt Spinning Apparatus

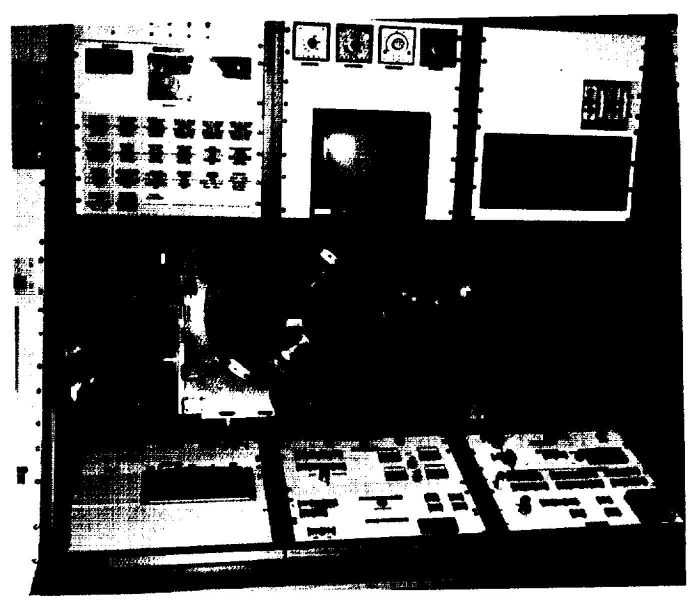
Rapid solidification, a relatively new materials process, offers advantages over conventional casting methods. These include finer grain size, greater chemical homogeneity, and more frequent metastable phase formation. Lewis has constructed two melt spinning rigs for rapid solidification. One, which uses the thermal mass of a solid wheel for cooling, is limited to short runs of about 20 m of ribbon. The other is essentially a continuous casting mill, incorporating a water-cooled wheel and designed to produce 500 m of rapidly solidified ribbon in 30 sec. Either controlled atmospheres or vacuum may be used. Both spinners include residual gas analyzers for the study of atmosphere effects. Because the process is so rapid, videotaping is used for observing the casting behavior and subsequent modeling research. Materials with melting points as high as 1700 °C can be rapidly solidified in these facilities.



Current interests include intermetallic, nickel-, iron-, and copper-base high-temperature alloys. Much of the rapid solidification research is performed in conjunction with colleagues from industry and academia.

Extrusion Press

Extrusion is a material working process which can be used to break down cast microstructures into elongated or equiaxed grains; to densify powders into solid, pore-free materials; and to produce structural shapes. Lewis has a large, vertical extrusion press capable of exerting pressures up to 1310 MPa. Materials can be preheated to temperatures as high as 2200 °C and extruded at speeds ranging from 0.02 to 50 cm/sec. Extrusion has been extensively used to develop high-strength, high-temperature-refractory-metal, oxide-dispersion-strengthened superalloys and is the process of choice to produce intermetallic aluminides from prealloyed powders.



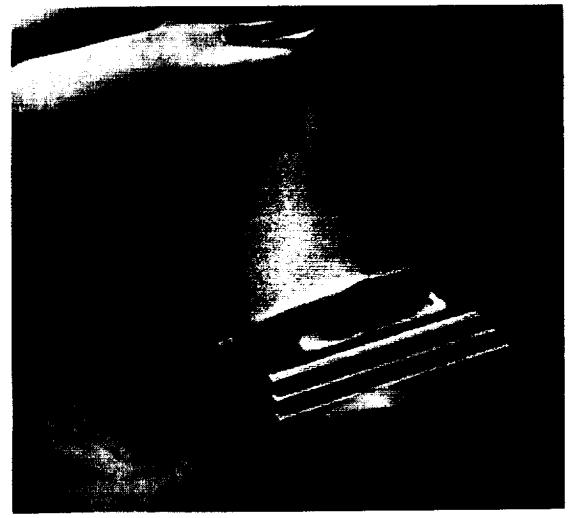
Rolling Mills and Swaging Machines

Both laboratory-scale rolling mills and swaging machines are available at Lewis for processing experimental materials. A two-high hot-rolling mill can handle alloys up to 6 cm thick and 20 cm wide while the precision cold rolling mill can be configured in two-, four-, and six-high modes, the latter being capable of producing foils 25 μm thick. Both hot and cold rotary hammering of bars can be conducted in the three swaging machines. Full sets of dies permit working of materials from 3 to 0.03 cm in diameter. These conventional metal-working techniques have been applied to a wide range of alloys, from low-temperature, high-strength steels to wire-reinforced superalloy composites in order to study the effects of secondary processing on mechanical properties.

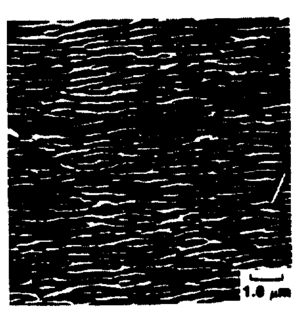
Single-Crystal Superalloy Research

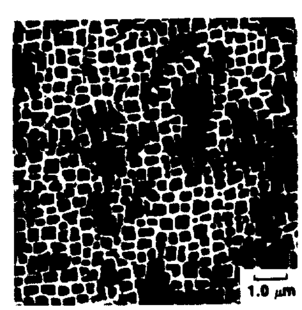
Research on single-crystal superalloys is directed toward understanding the effects of processing, composition, microstructure, and the environment on mechanical behavior. Both simple model alloys and commercial alloys are being studied. Of particular interest are the microstructural changes which occur under stress at high temperatures and the effects which composition and the initial microstructure have on these changes.

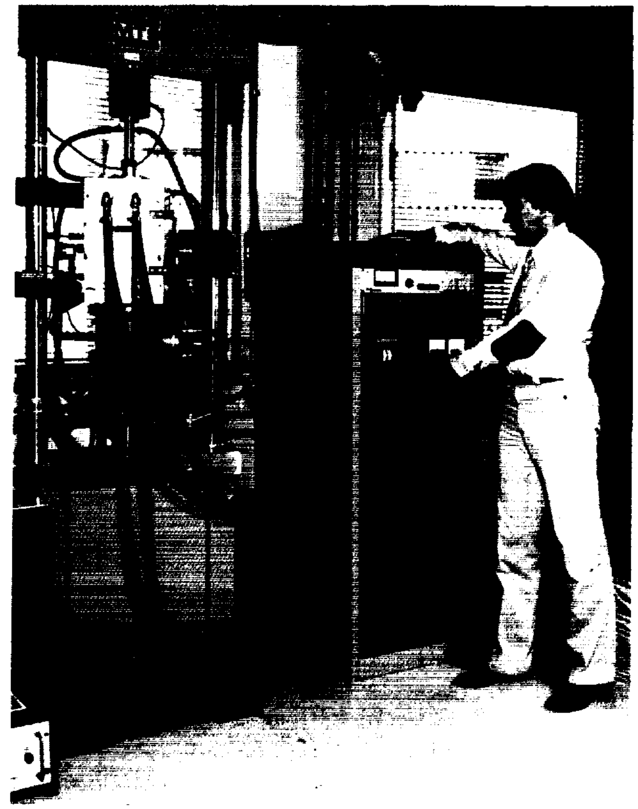
In some superalloy compositions, the strengthening precipitate coarsens perpendicular to the applied stress axis preventing dislocation climb around the particles. Such coarsening behavior is being studied by computer modeling.



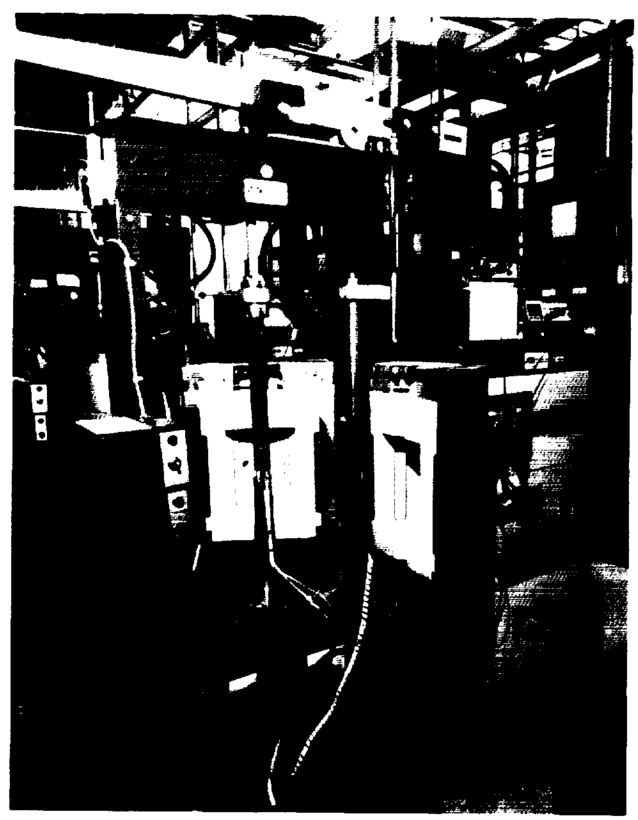
Directional solidification furnace.—This furnace is currently being used to grow single crystals by directional solidification. Molten metal is poured into a ceramic shell mold which is withdrawn from the hot zone at a controlled rate. As the mold is withdrawn, many grains grow in the direction of heat withdrawal. Of the several grains entering a geometrical constriction (note the cross-cut helix in the illustration), only one grain emerges. This grain solidifies as a single crystal through the remainder of the mold.







Mechanical fatigue.—The effects of alloy microstructures, crystal anisotropy, and gaseous environments on the strength and life of single-crystal superalloys are being studied. The conditions of cyclic temperature and stress simulate those occurring in gas turbine or space shuttle engines. This fatigue apparatus allows fatigue testing in air, inert gas, or vacuum as a means of separating environmental effects.



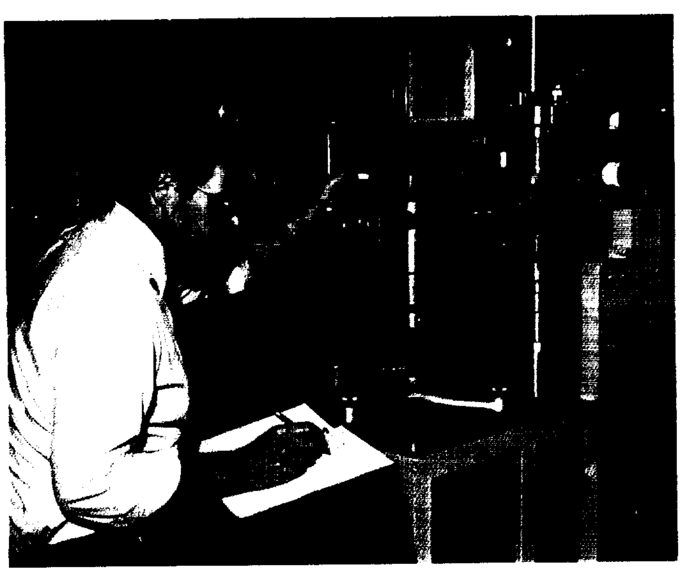
Intermetallic Compound Research

Research on the intermetallic compounds of iron aluminide and nickel aluminide focuses on alloying to improve the high-temperature strength and low-temperature ductility of these lightweight materials. Testing for compressive creep is done in the facilities shown. Compressive creep as a function of temperature and stress is used to determine the effects of various alloying additions on the strength of these advanced materials.

Materials for Space Power

The Materials Division at Lewis, in cooperation with other government agencies and private industry, is involved in materials research for advanced space power concepts as required to provide electrical power for the space station. Achievement of highly efficient power generation in space necessitates high operating temperatures for dynamic systems. Materials of interest to meet the requirements of high strength at high temperatures include the refractory metal alloys of niobium, tantalum, and molybdenum. Current research focuses on understanding the effects of alloy composition, heat treatment, and welding parameters on the long-term creep behavior of the refractory metal alloys. Other research efforts will help assess the long-term compatibility and corrosion of heat receiver salts and their containment materials to be used for solar dynamic electric power systems. Another program associated with advanced space power systems deals with the potential for mass transfer of oxygen, carbon, and nitrogen in circulating helium.

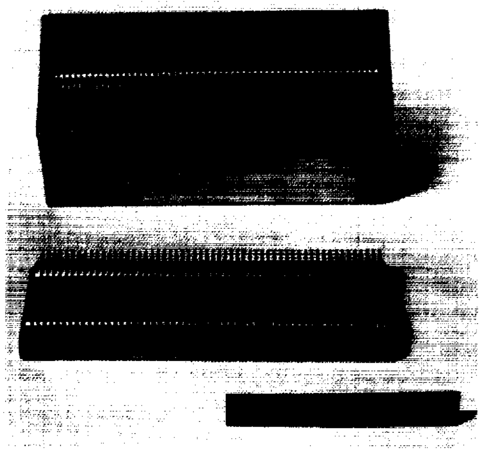
Ultra-high-vacuum creep.—The ability of high-temperature propulsion or power system materials to resist elongation or stretching under load at high temperature is of paramount importance to their usefulness. For ultra-high-temperature space power applications, this ability must be determined under conditions simulating the vacuum of space. For that reason, Lewis developed ultra-high-vaccuum creep facilities where materials can be tested to temperatures above 3000 °C. Creep-rupture, stress-rupture, and strain-rate-sensitivity tests as well as dislocation-interaction studies can be conducted without concern for the environmental contamination that would occur if the materials were tested in air.



Welding.—For the refractory metals and nickel-base alloys, gas tungsten arc welding (GTAW) is the most versatile process. GTAW is usually employed in the manual mode with argon or helium shielding. Lewis has a vacuum purge chamber suitable for use in the GTAW of refractory metals. Facilities for electron beam welding (EBW) are also available. The electron beam is a higher intensity heat source than is the GTAW torch. Thus, relatively narrow fusion welds (see insert) can be produced at low heat input by EBW. The vacuum environment used in EBW is also especially attractive to avoid atmospheric contamination of refractory metals.

Inert gas mass transfer.—In long-term electric power systems for space, transport of interstitial elements by the circulating helium working fluid may pose a problem. Experiments designed to address this potential problem were conducted in simulated space conditions.

Molybdenum capsules, containing Nb–1-wt-% Zr coupons, were filled with helium and heated in a vacuum for periods exceeding 1000 hr. The apparatus shown is equipped with a resistance-heated furnace within a vacuum chamber. This allowed the capsule to be positioned such that the bottom and top portions of the capsule could be maintained at different temperatures, 1800 and 770 K, respectively. The resulting temperature gradient assured helium circulation within the capsule. These coupons were evaluated for variations in nitrogen, oxygen, and carbon content.

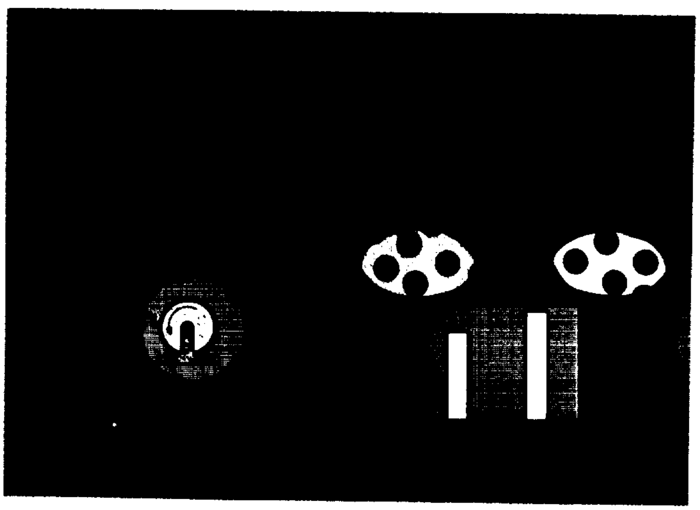


Solar dynamic heat storage system.—One design currently under consideration to provide continuous electrical energy for the proposed space station involves a latent heat storage system to be used in conjunction with a turbine-driven generator. A constant supply of energy can be provided to the engine by the melting and subsequent freezing of a salt. In support of this effort, studies have been initiated at Lewis to determine the effect of long-term exposure to molten salt and salt vapor on the mechanical properties of potential containment materials. These experiments utilize a "bread pan" capsule containing approximately 100 mechanical property test specimens and about 200 cm^3 of salt (see insert). Loaded capsules with an additional 50 test specimens, to determine the effects of annealing alone, will be subjected to temperatures slightly above the melting point of the salt for times from 100 to 10 000 hr.

Metal-Matrix Composites

Metal-matrix composites are made by incorporating high-strength, high-modulus fibers into metal matrices. This type of composite offers the potential of substantially reducing the weight of many engine components ranging from aluminum and titanium fan blades to turbine components. For example, fiber-reinforced superalloys offer a unique means of extending the high-strength properties now being achieved in directionally solidified castings to even higher temperatures by incorporating tungsten or ceramic fibers. These very-high-strength fibers can be oriented in such a manner as to achieve optimum properties in both the axial and transverse directions. The combination of tungsten fibers and superalloy matrices offers potential improvements in use-temperatures of approximately 150 °C over conventionally cast superalloys and 100 °C over the current directionally solidified materials being used for aircraft and rocket-engine turbine blades.

Copper-base alloys.—Improved rocket-thrust chamber life by using a metal-matrix composite material made up of tungsten wire embedded in a copper matrix is being evaluated at Lewis. High-strength copper-base alloys are currently used for constructing rocket-thrust chamber liners to transmit high heat loads from the hot-gas-side wall to a coolant while still providing sufficient strength to carry pressure loads. However, the severe environment to which the thrust chamber wall is subjected causes an irreversible deformation and thinning of the cooling passage wall during each cycle of operation. Repeated thermal cycles cause cracks in the cooling passages and eventual failure of the thrust chamber. The tungsten wire in the metal-matrix composite possesses the high strength necessary to carry the pressure loads and prevent deformation of the cooling passage wall while the copper matrix possesses the high conductivity necessary to transmit the heat load to the coolant. Laboratory data show that the tungsten fiber—copper composite has a rupture strength 80 percent higher than the currently used copper-base alloy and a thermal conductivity reduction of only 5 percent. Both cylindrical and contoured rocket-thrust chamber liners were fabricated using the tungsten fiber—copper composite material and are currently being evaluated for use in actual rocket-engine applications.



Arc-spray monotape fabrication.—A new method and apparatus have been developed for metal-matrix composite fabrication. The arc-spray fabrication facility sprays very clean liquid metal on an array of fibers which are wrapped on the drum surface (see fig.). The liquid metal is produced in an argon atmosphere by striking an electric arc between two metal wires causing the tips of the wires to melt. High-pressure argon sprays the liquid onto the array of fibers as shown in the figure. Sufficient metal is sprayed to cover completely the fibers and create a single raw fiber composite called a monotape. Monotapes have been produced up to 18 in wide and 47 in long. The system can produce very clean metal sprays of, for example, iron, nickel, and niobium alloys. Any continuous fiber may be used as a reinforcement. The most successful fibers used to date have been tungsten, molybdenum, and silicon carbide. The individual monotape plies are bonded together in a variety of orientations to produce large complex composites in a routine fashion. Standard techniques such as hot isostatic pressing (HIP) or hot pressing are used to fully densify and bond the monotapes into composite components.

Polymer Matrix Composites Research

The objective of polymer matrix composites research at Lewis is to develop technology for the new generations of polymer matrix composites intended for application in advanced aeropropulsion systems. Other applications for the newly developed technology are in airframe and space structures. To achieve this objective, research is performed in the following areas: monomer/polymer synthesis, polymer/composites characterization, polymer/composites processing, environmental effects, and thermomechanical properties. Emphasis is given not only to developing improved materials but also to achieving a fundamental understanding of materials' behavior at the molecular level. The matrix materials currently under investigation are high-temperature-resistant polymers such as condensation and addition-cured polyimides and ladder types of nonheterocyclic polymers. Graphite, glass, boron, and aramid fibers are used as reinforcing materials.

Polymer Synthesis

Well-equipped polymer laboratories are available in which the synthesis of high-temperature matrix resins and adhesives is performed.

Polymer Characterization

The performance limits of a composite depend on the properties of both the matrix polymer and the fibers. Lewis has a variety of instrumentation for determining the chemical structure of polymer resins and for measuring the physical and mechanical properties of composites. Chemical characterization is performed with a Fourier transform nuclear magnetic resonance spectrometer, a Fourier transform infrared spectrometer, an ultraviolet spectrometer, a gas chromatograph, several liquid chromatographs, and a thermogravimetric analyzer. Physical and mechanical properties are measured with differential calorimeters, thermomechanical analyzers, a dynamic spectrometer, and a dielectric spectrometer.

Computer Modeling

The SYNCHEM and LHASA programs are expert systems used for computer-assisted organic synthesis. These programs, working with a library of information about known chemical reactions, attempt to devise a synthetic pathway to a target molecule specified by the chemist. These programs often suggest a series of reactions that are unexpected. Their major advantage is that they can suggest unusual, but valid, pathways that a chemist might not normally consider. Primarily used in the pharmaceutical industry, SYNCHEM and LHASA are being applied at Lewis to the synthesis of monomers for new polymer materials.

Composites Processing

Extensive facilities are available for fabricating and evaluating polymer matrix composites. Test panels fabricated in these laboratories are used to evaluate the performance characteristics of the newly synthesized polymers as matrix resins. To fabricate the test panel, a single layer of fibers impregnated (wetted-out) with the polymer under investigation is first cut into pieces, called plies, with the fiber orientation as specified by the design engineer. The desired number of plies are then stacked in a predetermined sequence into a matched metal die and cured under heat and pressure in a press or an autoclave.

The autoclave shown provides an automatically controlled pneumatic pressurization system and a programmable heating cycle. Pressures up to 17 atmospheres and temperatures to 400 °C are possible. Usable work space within the autoclave measures 1 m in diameter by 1 m in length.

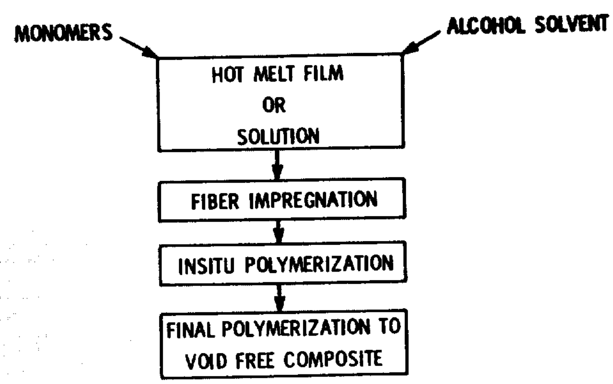
Composite Evaluation

Extensive facilities are available to determine the mechanical properties of the composites over a broad temperature range (−320 to 700 °F). The tensile, compressive, flexural, and shear properties of the newly developed composite materials are determined to establish the potential of these materials for meeting end-use requirements. Research is being conducted to develop improved materials and also to achieve a fundamental understanding of materials behavior at the molecular level.

The drop weight test apparatus is a unique test facility being used to generate data to correlate polymer properties with composite properties. With this facility, the low-velocity impact resistance of composite or polymeric materials can be measured at temperatures between −75 and 125 °C. The test equipment is currently being used to extend the knowledge of relationships between polymers and composite toughness to include the effects of polymer molecular structure and structural changes on composite impact response.

PMR Polyimide Technology

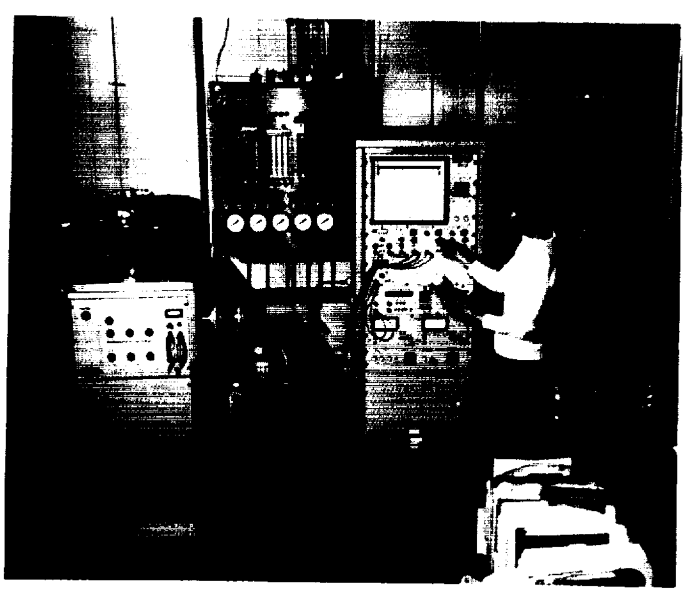
In response to the need for a processable higher-temperature-resistant polymer matrix material, investigators at Lewis developed the novel class of polyimides known as PMR (polymerization of monomer reactants) polyimides. Fiber-reinforced PMR polyimides have superior processing characteristics and exhibit excellent retention of mechanical properties during continuous use at temperatures up to 600 °F. The PMR concept has been adopted by other investigators, and prepreg materials based on the PMR polyimide designated as PMR–15 are commercially available from the leading suppliers of composite materials. The excellent properties, processability, and commercial availability of PMR–15 composites have led to their acceptance as viable engineering materials for application in engines, space structures, and weapon systems. They are being used to fabricate a variety of components from small compression molded bearings to large autoclave molded engine ducts. An example of an engine component fabricated from T300 graphite fabric and PMR–15 is shown. The composite duct, located above the carriage, is installed on a F404 test engine. The composite duct was developed by General Electric under a program sponsored jointly by the Navy and NASA Lewis. Substitution of the composite duct for the original titanium part results in a 15-percent weight savings and a 35-percent cost reduction.



Ceramic Materials Research

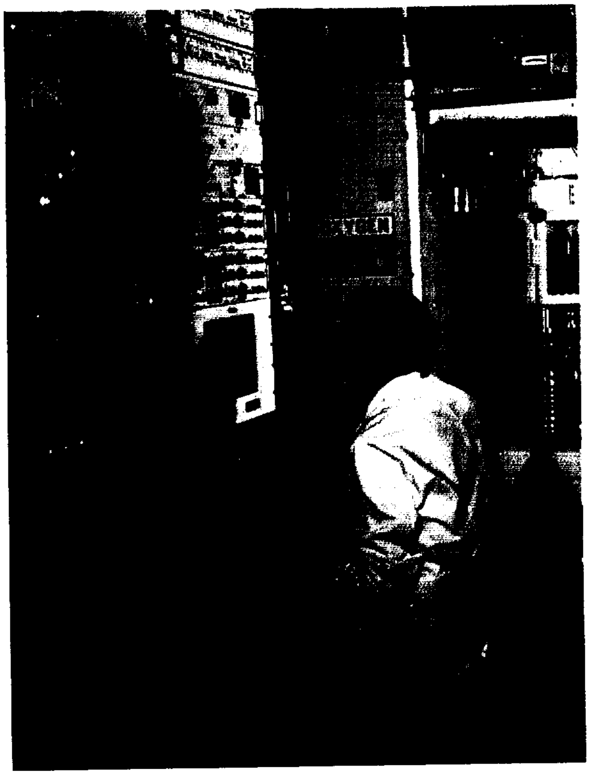
Advanced aerospace propulsion and power systems can often benefit from the application of ceramic materials with higher temperature capability. One objective of ceramic research at Lewis is to identify ceramics and ceramic composites with the required strength, fabricability, reliability, and durability for use in aerospace systems. The emphasis of the research is on basic understanding of processing, i.e., microstructure-property relations in high-temperature structural ceramic systems.

A second objective is to understand and model the role of microgravity in order to explore the benefits of containerless ceramic processing for a broad range of ceramic materials. Benefits are sought from the identification of high value-added materials for production in space and from the enhanced understanding of ceramic processing which can lead to more effective ceramic processing on Earth.



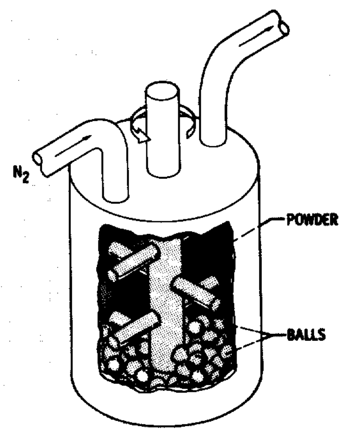
Ceramic Processing

A new ceramic processing and characterization laboratory featuring a filtered positive-pressure environment and near "clean room" construction is in operation at Lewis. All ceramic powder handling and characterization from the as-received powders through green consolidation can be accomplished with minimum contamination. This facility is used for research directed toward improved reliability, toughness, and strength in advanced structural ceramics and ceramic matrix composites. Available equipment permits particle size classification, slip characterization, comminution (ball mill, vibratory mill, and attrition mill), wet and dry shape making, and cold isostatic compaction. Also available is a state-of-the-art simultaneous thermal analyzer with thermogravimetric and differential thermal analysis capabilities to 2400 °C and dilatometry to 1600 °C.



Ceramic Attrition Mill

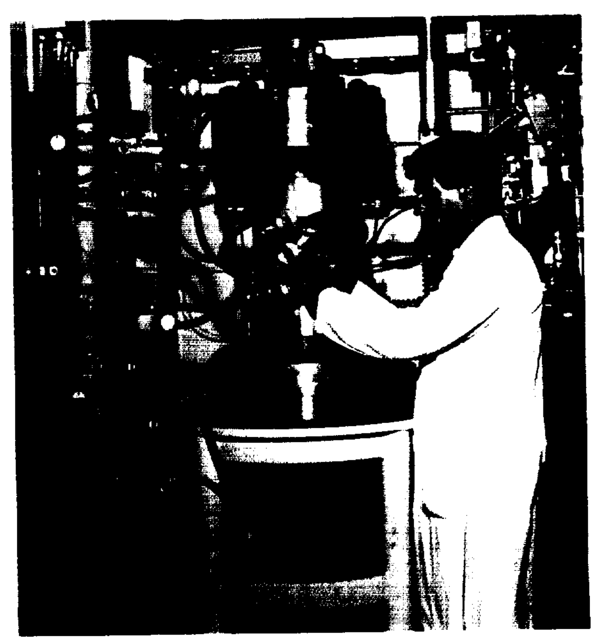
Unique all ceramic (SiC and Si_3N_4) attrition mills have been developed both to grind starting ceramic and additive powders into very small particle sizes and to assure the intimate mixing of such powders. The use of ceramic hardware minimizes the incorporation of detrimental impurities, and the fine particle sizes achieved enhance sinterability. These factors are important in the production of low-cost structural and rotating ceramic components for applications in the hot sections of future heat engines (turbine, Stirling, etc.) for terrestrial and aerospace applications.

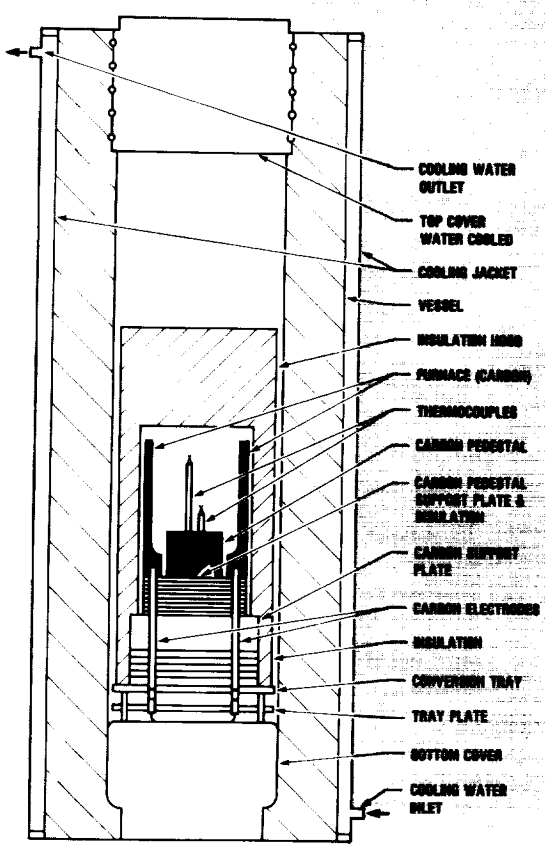


Shaping and Fabrication

Many of the materials being studied for advanced power and propulsion systems must operate at very high temperatures. Because this requirement makes their fabrication at conventional processing temperatures and pressure difficult, unique facilities have been developed to shape these materials.

High-pressure sintering furnace.—High-strength, high-temperature silicon nitride ceramics are difficult to fabricate into useful shapes. While high-temperature sintering at atmospheric pressure promotes densification, decomposition of the silicon nitride takes place under such conditions. Exposure to nitrogen pressures greater than 60 atmospheres during heating, however, allows sintering temperatures approaching 2150 °C to be used. In this way, decomposition can be avoided and nearly 100-percent-dense shapes can be obtained.





Hot isostatic press.—Many of the advanced materials are difficult and costly to machine once they have been consolidated. Thus, hot isostatic pressing is used to consolidate powders of metals and ceramics to near final shape. Combining gas pressures of over 1300 atmospheres and temperatures up to 2200 °C makes possible the production of both metal and ceramic parts in fully dense form. Previously cast or sintered components can also be pressure and temperature treated to heal microscopic flaws and thus further reduce the potential of early mechanical failure. Such a process is ideal for fabricating ceramic turbine airfoils and integrally bladed turbine rotors for automotive gas-turbine service. It also has the potential for forming other unique combinations of metals and ceramics into complex shapes which would be more difficult and/or extremely costly to fabricate by more conventional consolidation and machine procedures.

Composites

A variety of facilities exists for characterizing and processing fibers used as reinforcements for resin matrix, metal matrix, carbon matrix, and ceramic matrix composites. Facilities are also available for fabricating fiber-reinforced monotapes and consolidating them with other precursors into these various composites for evaluation of composite strength and durability.

Fiber testing.—A variety of characterization and testing facilities exists for measuring the physical, chemical, and mechanical properties of fibers before and after subjection to the environmental conditions typically encountered during composite consolidation and use. Unique among these are high-temperature fiber test and processing equipment and dynamic property measurement equipment. If resistance heating is used, the high-temperature test rigs are capable of measuring fiber modulus, strength, creep, and thermal expansion for temperatures up to 1500 °C and for various environmental gases under controlled pressure conditions. The dynamic property equipment uses specimen mechanical resonance to measure the temperature dependence of such properties as dynamic modulus (flexural and torsional), internal friction, and transverse thermal conductivity. Working with reinforcements as small as a few micrometers in diameter or bulk composite materials as large as $0.2 \times 1.0 \times 10$ cm in size, one can use these facilities to generate practical dynamic property data which can, in many cases, also be used for nondestructive evaluation of material microstructure.

Hot press.—One proven method for consolidating fiber-reinforced composite materials is by alternatively stacking matrix foils and fiber mats and then uniaxially pressing the stack under conditions in which the matrix flows between the fibers and sinters. Hot press facilities for high-temperature metal and ceramic matrix composites allow this type of processing under the extreme fabrication conditions required for these materials. For example, when induction-heated graphite dies are used, temperatures as high as 1700 °C are obtained under consolidation pressures up to 5000 psi. These facilities which can be operated under vacuum or controlled environments permit the fabrication of 10 in^2 rectangular billets with lengths up to 5 in. These lengths allow subsequent fabrication of test specimens suitable for composite tensile testing both at low and high temperatures.

Static fatigue.—For materials with envisioned service at high temperature, an important consideration is the reduction in structural performance caused by an adverse combination of mechanical loading and aggressive gases such as oxygen. To examine this effect for advanced composite materials, static fatigue facilities are available in which modulus of rupture specimens can be flexurally loaded for extended time periods under controlled environments and isothermal conditions up to 1500 °C. This capability of testing specimens with lengths to 30 cm is especially important for ceramic and carbon matrix composites.

Thermal shock test rig.—Lewis facilities for testing materials and coatings under high heat flux conditions include a plasma torch and a hydrogen-oxygen thermal shock test rig. The plasma torch can provide extremely high local heat fluxes by generating flame temperatures in excess of 3000 °C. The thermal shock test rig shown is capable of producing pressures up to 600 psi, a range of hydrogen to oxygen ratios, and gas temperatures to 1650 °C. A variety of specimen sizes and shapes can be used to determined the durability of materials exposed to repeated thermal stress.

Environmental Durability

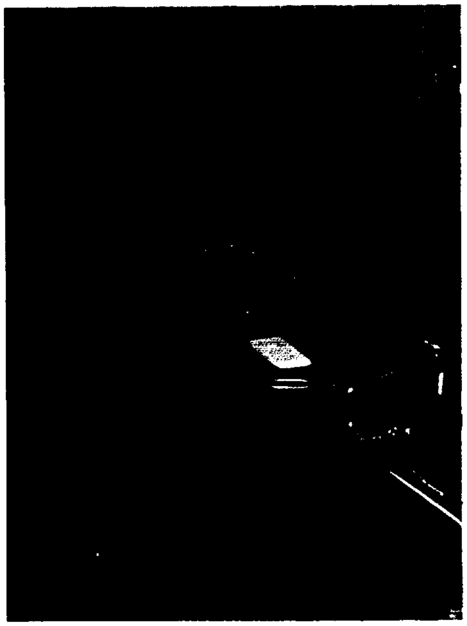
High-temperature environmental durability is a prerequisite for present and future propulsion and power systems. Minute qualities of certain impurities in the combustion air or fuels burned in advanced heat engines can lead to material degradation of high-temperature components. Such environmental attack can directly limit the life of components, or it can cause material wastage which reduces the load bearing area to the point where mechanical failure occurs prematurely. A broad range of research activities is required in order to attain a sufficient understanding of environmental attack to facilitate methodologies for alleviating such attack and its adverse effects. These activites include work on the fundamentals of flame chemistry, studies of mass transport and deposition of corrosive species contained in the products of combustion, investigations to elucidate the mechanisms of oxidation and corrosion attack, and development of strategies and coating systems to provide high temperature environmental resistance. Investigations encompass a broad range of materials, e.g., superalloys, ceramics, and composites as well as metallic and ceramic coatings.

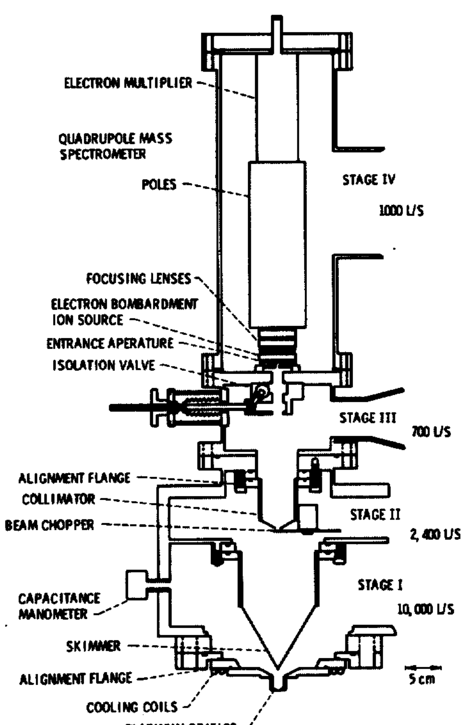
High-Pressure Mass Spectrometric Sampling System

Predictions of the types of compounds that can form in combustion processes require an understanding of the thermodynamics and kinetics of flames. To identify the chemical species formed in appropriate combustion flame systems, Lewis scientists developed a unique sampling system whereby systems at total pressures up to 1 atmosphere can be studied while maintaining the chemical and kinetic integrity of the gaseous species. The sampler has been used to identify and measure concentrations of reactive species, condensates, free radicals, and reactive intermediates in certain flame and gaseous flow reaction systems. With this facility, thermodynamic and kinetic data are being obtained to provide improved insight into the chemical reactions and mechanisms involved in oxidation, hot corrosion, and gas-solid interactions of materials used in practical engineering devices.

Cyclic Oxidation Test Facilities

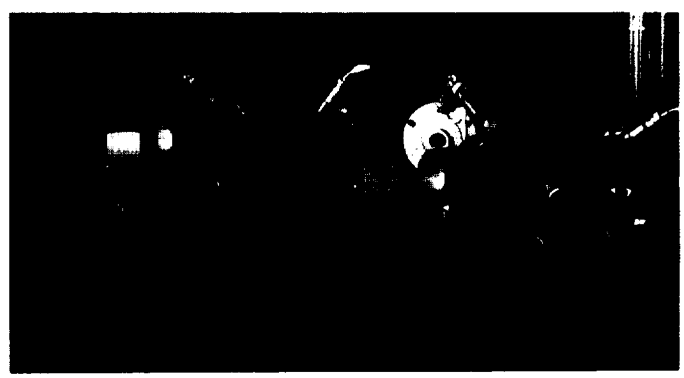
In aircraft propulsion systems, high-temperature components are subjected to cyclic temperature exposure. Most materials respond to cyclic temperature regimes differently than they do to isothermal conditions. Lewis engineers have developed a cyclic furnace test facility to study the oxidation of materials over a broad range of temperature and cycle frequency. Experimental data obtained in this facility have contributed to the development of a model capable of predicting cyclic oxidation behavior. The studies at Lewis have led to an increased understanding of oxidation phenomena and the development of environmentally durable oxidation-resistant alloys.

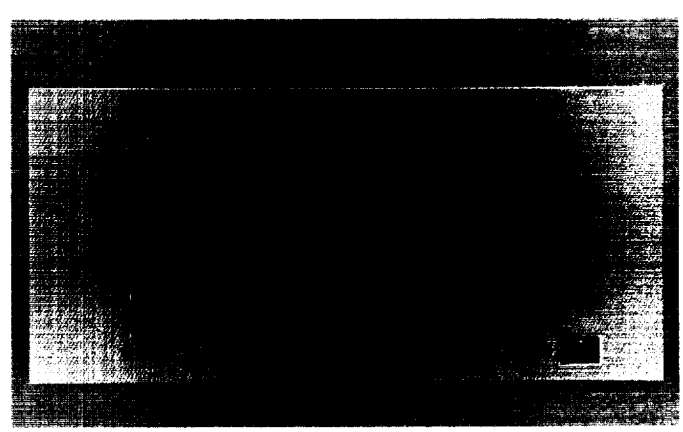




High-Velocity Oxidation, Hot-Corrosion Test Facility

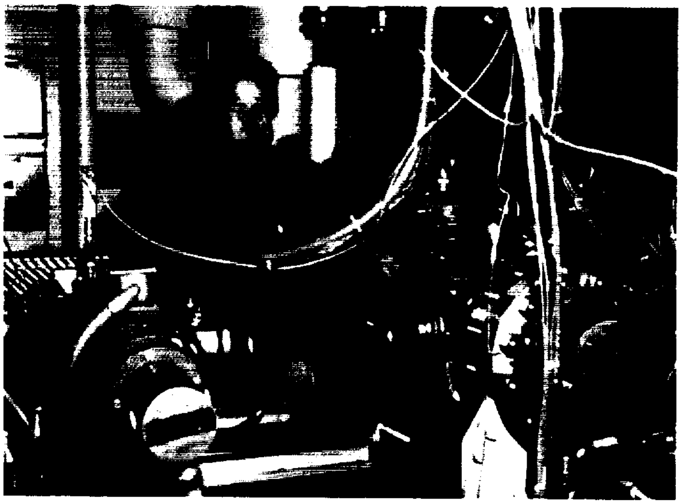
Small Mach 0.3 combustion rigs provide the first step in engine environmental simulation. These rigs, in addition to the Mach 1 (sonic velocity) rigs, are used to establish the potential oxidation, corrosion, and erosion resistance of alloys and developmental high-temperature coatings. Because these rigs use very little fuel, they provide a highly efficient way to gain insight into potential engine behavior prior to actual engine testing. They are also used to verify attack models for engine-component life prediction. These rigs operate at temperatures to 1200 °C and the flames can be doped with a wide variety of possible fuel contaminents, e.g., sodium, potassium, chlorine, and vanadium. Forced-air cooling of the test specimen provides simulation of engine thermal gradients under startup, cruise, and shutdown conditions.

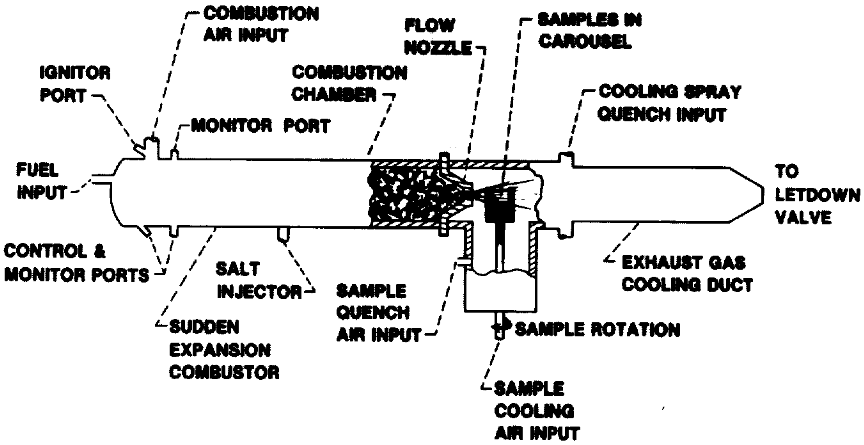




High-Pressure Burner Rig

This unique materials test facility provides the capability of testing at elevated pressures characteristic of those in advanced turbine engines. Furthermore, the heat flux available in this rig more closely duplicates that which exists in many heat engine applications. It is expected that sample temperatures near 3000 °C will be attainable with pressures up to 50 atmospheres and gas flow velocities of Mach 1.0. The rig burns jet fuel and air while the combustion products can be seeded with suitable compounds to produce flame effluents to simulate various environmental conditions encountered in advanced propulsion and power systems. Lewis researchers use this facility to investigate the high-temperature environmental durability of candidate materials for advanced aircraft.





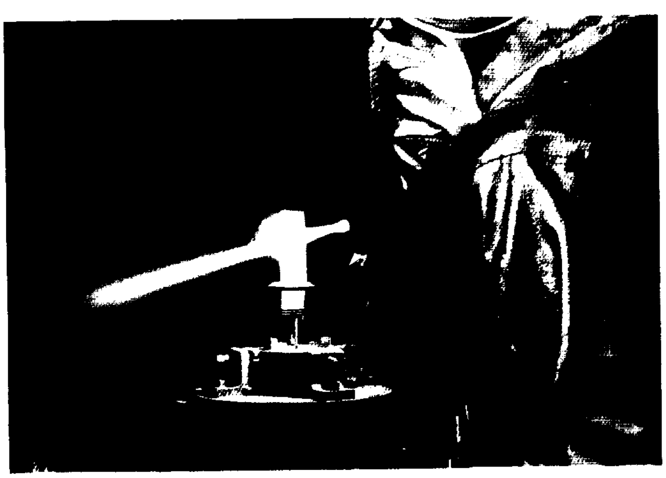
Scanning Electron Microscope

The scanning electron microscope is just one of the many sophisticated instruments used to study materials that have been exposed to environmental attack. With such tools, Lewis scientists and engineers have been able to characterize material attack and determine the fundamental processes involved in such an attack. Knowledge such as this is required to develop materials with improved environmental durability.

Coatings—Advanced aircraft and electric-utility gas turbines operate at very high temperatures and require extremely long component lives and resistance to combustion gases containing impurities that produce hot corrosion and erosion-producing impurities. One way to extend the life of high-temperature power and propulsion components is to apply protective coating materials that are more environmentally resistant than are the high-strength structural alloys. Simple single- or double-element coatings, although successfully employed in the past, cannot meet these needs. Thus, new coatings and deposition techniques are required. Such coatings can be applied by a number of techniques, including plasma spraying, physical vapor deposition, sputtering, ion plating, metallic cladding, etc.

Plasma spraying.—Flowing an inert gas through a high-power arc produces an extremely hot and yet nonreactive plasma "flame" into which powders of metals, ceramics, or mixtures of the two can be injected. These powders are heated to their melting points and are sprayed onto the surface of a part to be protected. Such spraying can rapidly provide a relatively thick coating whose properties are tailored to the specific powder system needed.

An 80-kW low-pressure plasma-spray facility is available with part manipulation capability to allow the uniform, reproducible deposition of coatings with nearly 100-percent density and negligible oxide impurities. Also available is a 40-kW plasma-spray facility for developing advanced coating systems. With the aid of such facilities, Lewis is researching plasma-sprayed metallic and ceramic thermal-barrier coatings having the potential for lower cost and more compositional flexibility than may be possible by any other approach.



Alternative coating deposition processes.—Lewis has capabilities in both physical vapor deposition and magnetron sputtering so that metals, alloys, intermettalic compounds, and ceramics can be deposited in layers as thin as a few micrometers. Sputter deposition, for example, employs high-velocity argon atoms which bombard the target and dislodge ions of the target material. These ions migrate across an electric field and deposit on a suitable substrate. The primary purpose of work in these areas is to produce coatings which have improved environmental resistance or which modify the surface of materials with surface flaw sensitivity, thereby improving their strength and wear resistance.

Facilities are also available for electrodeposition, chemical vapor deposition by the pack cementation process, and deposition from the gas phase at high temperatures or by using a plasma assist.

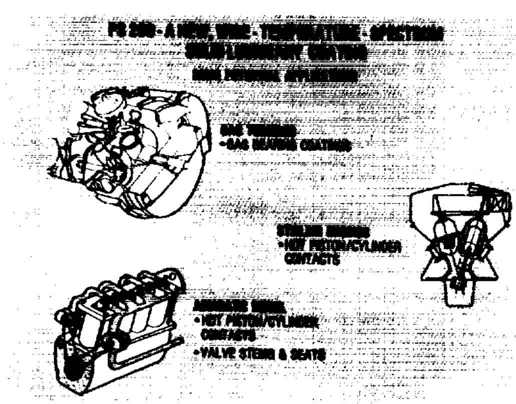
CO_2 laser.—A 300-W, continuous-wave CO_2 laser is available for surface processing (i.e., surface melting and testing) of ceramic or metallic materials and coatings. Flat and cylindrical specimens can be processed in air or in protective or reactive atmospheres. The maximum obtainable power density is about 3×10^4 W/cm^2.

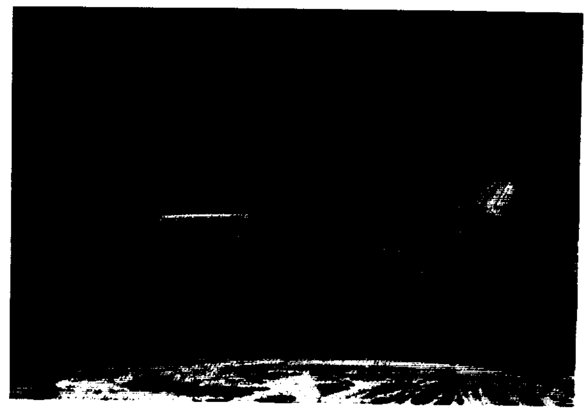
Surface Science

One area of research currently being conducted at Lewis is the study of solid surfaces and their interactions with environments and other solids. The interactions include studies in oxidation, corrosion, adhesion, friction, wear, lubrication, and interfacial behavior. The behavior of interfaces in solid to solid contact is extremely important in tribology, in electronic packaging, and in such mechanical systems as heat engines, aircraft components, and space mechanisms. The research activity is at the basic level with a host of analytical surface tools being employed to help characterize surfaces. A number of these surface tools are incorporated directly into the research experimental devices to facilitate in situ surface studies.

Tribological Experiments

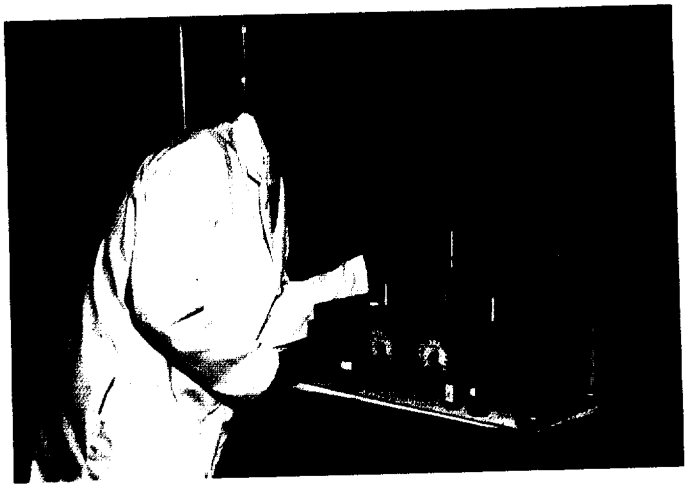
Ceramic materials have become an integral part of such mechanical components as advanced heat engines. The tribological behavior of these materials at temperatures to 1200 °C is extremely important. The high-temperature friction and wear apparatus allows the researcher to measure the friction and wear properties of ceramics over a broad temperature range.

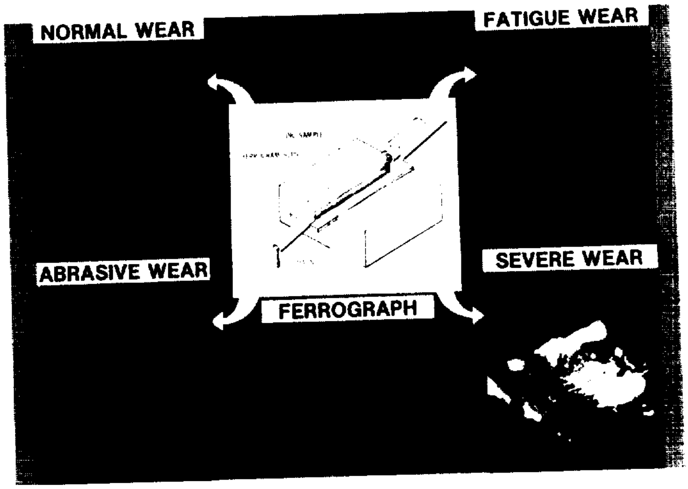




Solid Lubricants

Advanced designs of the adiabatic diesel, the Stirling engine, and gas turbine engines all require high-temperature lubricants. In many cases the temperature and the chemical reactivity of the atmosphere are too extreme for oils or greases to be used and solid lubricants must be considered. Conventional solid lubricants such as molybdenum disulfide and graphite are useful to maximum temperatures of 350 to 500 °C. For even higher temperatures, novel solid lubricants must be used. In a research program at Lewis to develop high-temperature lubricants, a plasma-sprayed composite coating was developed which is lubricative in chemically reactive atmospheres such as air or hydrogen from room temperature to 900 °C. The coating composition consists of hard, wear-resistant chromium carbide with additions of barium fluoride—calcium fluoride eutectic and silver. The additives act synergistically to reduce the friction and the abrasivity of the carbide while retaining its excellent wear resistance.



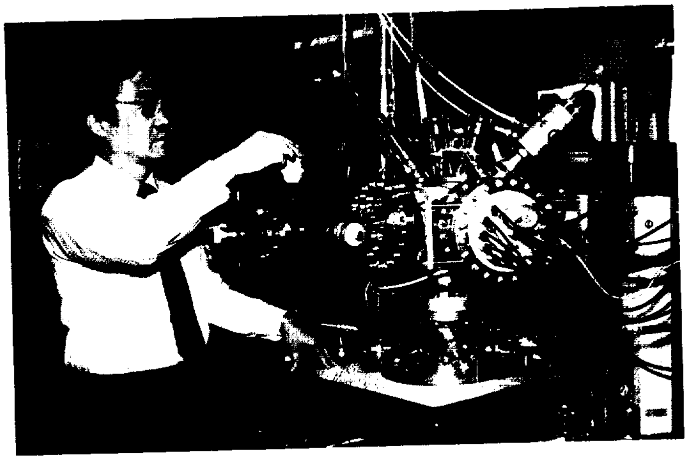


Ferrography

Frequently when two materials are in rubbing contact in such mechanical components as bearings, gears, or seals, wear debris is generated and carried into the fluid. The ferrograph permits the researcher to separate the debris from the fluid by magnetic means and to identify the wear mechanisms creating the debris.

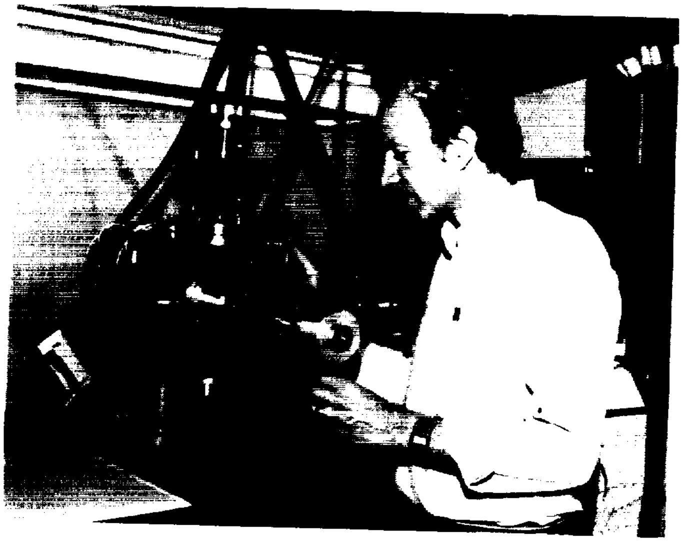
Chromatography

Research currently being conducted at Lewis involves the development of new lubricant molecular structures for advanced high-temperature lubrication. These new molecular structures must withstand thermal and oxidation degradation above 300 °C. Both liquid and gas chromatography are employed to assist researchers in the detailed analysis of the breakdown of new molecular structures as a result of thermal and oxidative degradation.



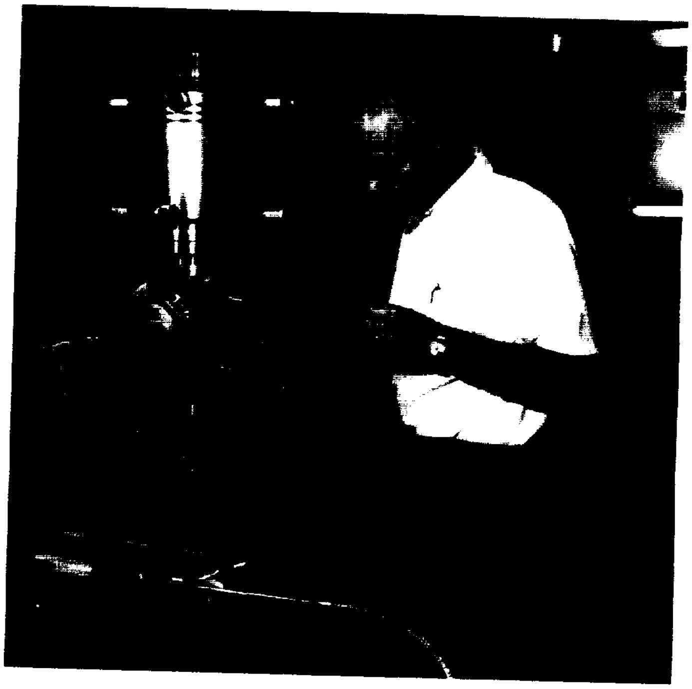
X-Ray Photoelectron Spectroscopy (XPS)

The XPS system incorporates both ion depth profiling capabilities and a device for conducting friction and wear experiments. Continuous measurement of the changes in surface chemistry during tribological studies is possible with this system. The ion gun allows analysis of both the surface and subsurface layers.



Scanning Auger Microscopy (SAM)

Frequently it is desirous to know the concentration of extremely small amounts of elements on a solid surface as well as being able to identify their locations on that surface. The SAM permits the researcher to identify all elements present on the solid surface, except for hydrogen and helium, to within a 0.01 monolayer sensitivity. It is used for oxidation, corrosion, and tribological studies.



Thermal Desorption Spectroscopy (TDS)

The TDS apparatus provides the capability of examining a solid surface over a broad range of temperatures. For example, a species can be absorbed at 4 K on a solid surface which is then allowed to warm. The temperature of desorption of the absorbate from the surface can be measured by using the x-ray photoelectric spectroscopic capability of the instrument. These measurements can be used in the calculations of bonding energies.

Scanning Electron Microscopy (SEM)

The SEM, with energy-dispersive x-ray analysis capabilities, is used to identify the topography and surficial chemistry of solids. The SEM is also used in the characterization of solid surfaces and the changes that occur microscopically in those surfaces as a result of oxidation, corrosion, or solid-state contact with other materials.

Materials Characterization Laboratories

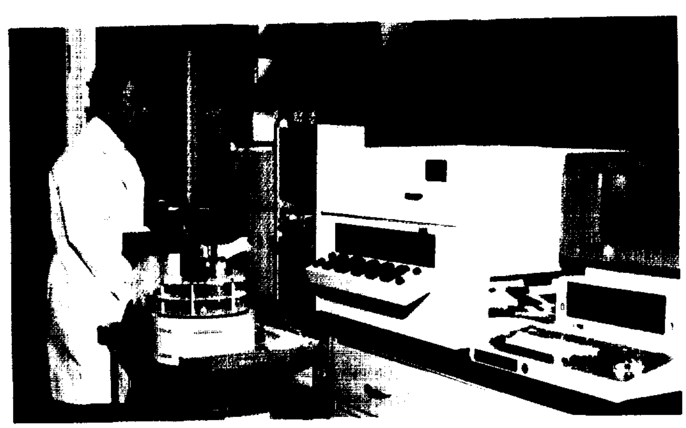
The Materials Division research activities are supported by characterization laboratories which provide analytical information as a research service to the aerospace materials scientists and engineers. The laboratories possess a wide range of analytical instrumentation that help the researchers gain a bettei understanding of both the composition and microstructure of advanced materials.

Chemical Characterization Laboratory

The analytical capabilities in the Chemical Characterization Laboratory are directed primarily at inorganic analysis of solids and liquids. The chemical methods of analysis and variety of analytical instruments available make possible the qualitative and quantitative determination of elemental constituents from trace levels (as low as parts per billion) through major components of a material.

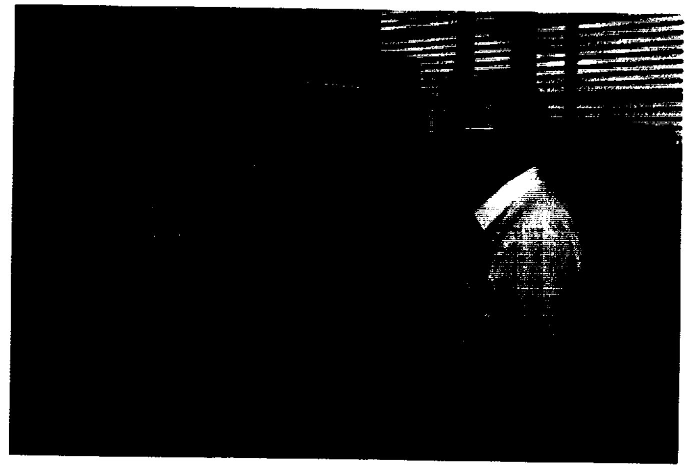
Inductively coupled plasma (ICP).—The inductively coupled plasma is a high-temperature excitation source for atomic emission analysis. The direct reading spectrometer allows simultaneous multielement detection of 34 elements.

Atomic absorption spectrometer (AAS).—The flame atomic absorption/emission spectrometer can sequentially determine a great number of elements quantitatively. This instrumental method is primarily used for determining traces to minor component levels of a sample.



X-ray fluorescence spectrometer (XRF).—X-ray fluorescence spectrometry uses high intensity x-rays striking the sample to excite the atoms of the component element. This technique can detect elements from sodium increasing in atomic number to uranium.

Gas analysis.—The gas analysis furnaces are used to determine interstitial carbon, hydrogen, nitrogen, oxygen, and sulfur in research materials. The oxygen determinator can quantitatively determine up to 20-percent oxygen using the inert gas fusion method.



Computer Resources

Computer resources provide Materials Division personnel with sophisticated data reduction methods for analyzing experimental data. Software specialists work to produce the best available computational techniques and to create communication links between division computers. Terminals and personal computers also give access to Lewis mainframes: IBM 3033 running TSS/370, two Amdahl 5840's running VM/CMS and MVS/XA, and the new Cray X-MP with two CPU's and 4 million words of main memory.

Microstructural Characterization

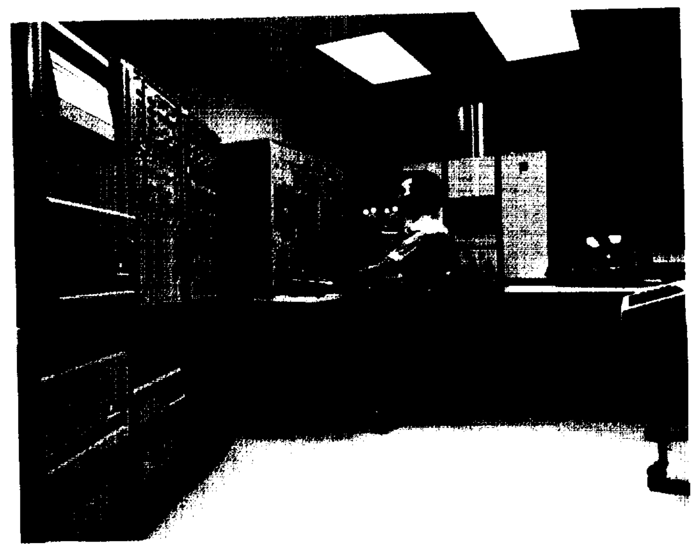
The Microstructural Characterization Laboratory provides analysis and documentation of the structure and composition of both metals and nonmetals. A variety of equipment is available for characterization including a complete metallographic laboratory, a well-equipped x-ray diffraction laboratory, and an electron-optics laboratory with four scanning electron microscopes, three transmission electron microscopes, and an electron probe microanalyzer.

Scanning electron microscope (SEM).—The scanning electron microsope is a basic tool for materials characterization. It offers magnifications up to 100 000X with good resolution and a depth of focus many times that of optical microscopy. When equipped with an energy dispersive spectrometer (EDS), it can also provide qualitative and quantitative elemental analysis.

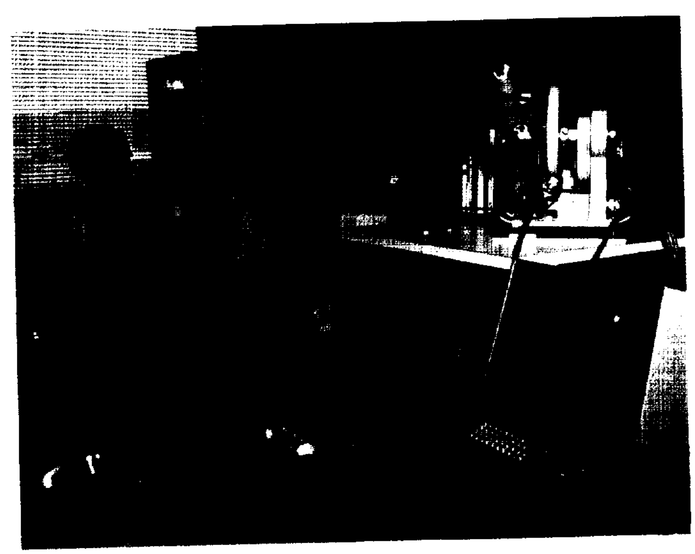
Transmission electron microscope (TEM).—The transmission electron microscope is essential to the basic understanding of materials behavior. This instrument can provide direct magnifications up to 480 000X and 0.2-nm resolution in the lattice imaging mode. Information can be obtained on microstructure, crystal structure, dislocation interactions, and chemistry.

Image analyzer.—The image analysis system is capable of measuring and statistically analyzing features directly from an image of a material's microstructure. It can provide a quantitative description of the amounts of various phases present and also yield information to be used in modeling the relationship between microstructure and properties.

Electron probe microanalyzer (EPM).—The electron probe microanalyzer can provide compositional information on small volumes (1 μm^3) of materials in a solid. It can provide quantitative analysis of elements with atomic numbers of 5 and above with an accuracy approaching 2 percent of absolute for up to 30 elements simultaneously.

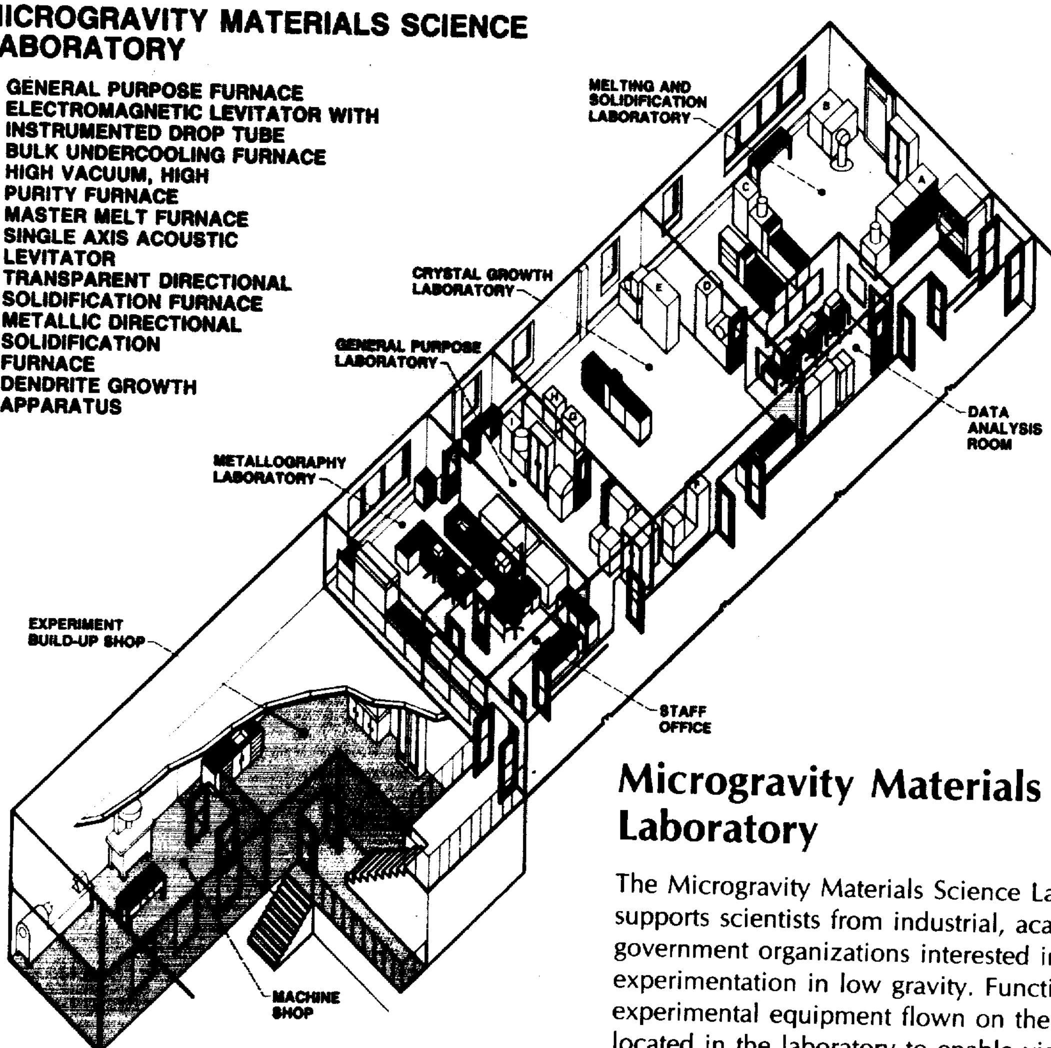


X-ray diffraction (XRD).—X-ray diffraction techniques can provide a wealth of information on the structure and properties of crystalline materials, including qualitative and quantitative phase analysis, lattice parameters, crystal structure, and orientation.



MICROGRAVITY MATERIALS SCIENCE LABORATORY

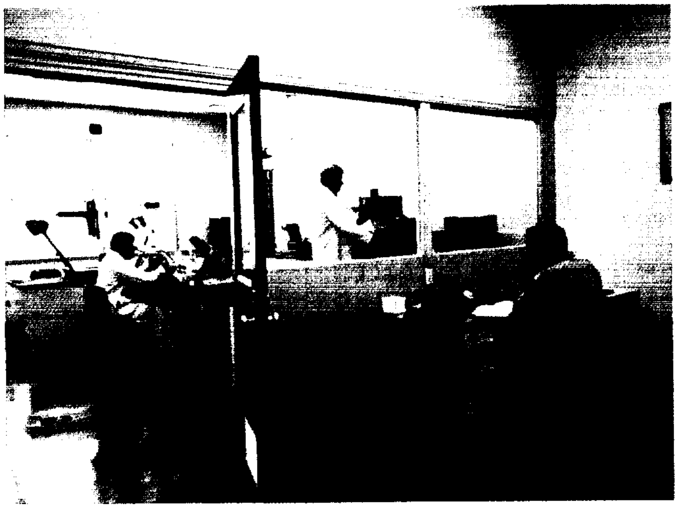
A. GENERAL PURPOSE FURNACE
B. ELECTROMAGNETIC LEVITATOR WITH INSTRUMENTED DROP TUBE
C. BULK UNDERCOOLING FURNACE
D. HIGH VACUUM, HIGH PURITY FURNACE
E. MASTER MELT FURNACE
F. SINGLE AXIS ACOUSTIC LEVITATOR
G. TRANSPARENT DIRECTIONAL SOLIDIFICATION FURNACE
H. METALLIC DIRECTIONAL SOLIDIFICATION FURNACE
I. DENDRITE GROWTH APPARATUS

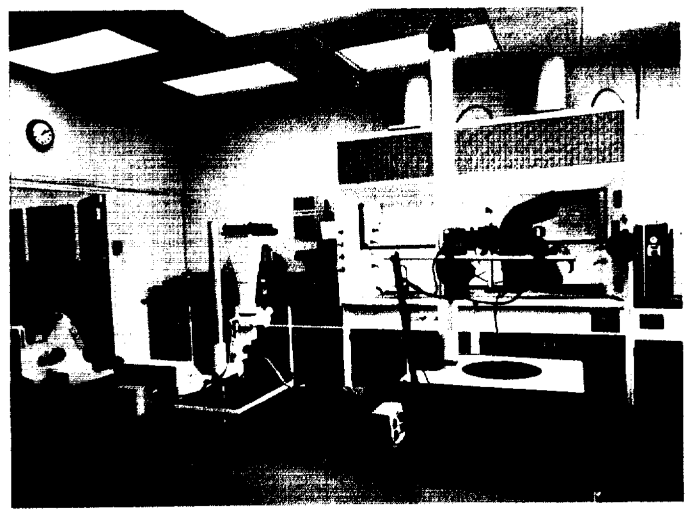


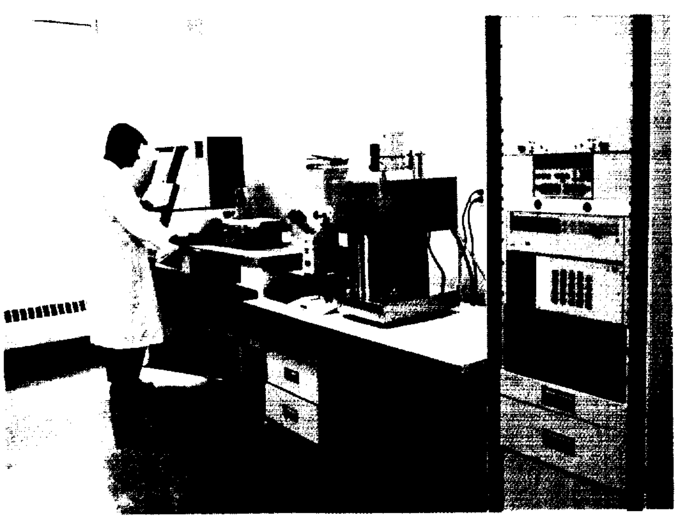
Microgravity Materials Science Laboratory

The Microgravity Materials Science Laboratory (MMSL) supports scientists from industrial, academic, and government organizations interested in materials science experimentation in low gravity. Functional duplicates of experimental equipment flown on the space shuttle are located in the laboratory to enable visiting scientists to better understand their experiment in a "1-g" environment using equipment configured to simulate in-flight hardware. The initial focus of the MMSL is on metal and alloy solidification and crystal growth research. Capabilities are being expanded to provide experimental equipment for low gravity research in polymers, ceramics, and glasses.

A scientist may apply to NASA Lewis Research Center to use the MMSL at little or no cost to explore the potential of a microgravity-related experiment before establishing a formal research program at his/her own organization. The experimental, characterization, and computational capabilities available through the MMSL afford visiting scientists the opportunity to develop an understanding of how low gravity affects materials and processing techniques.







General Purpose Furnace

The general purpose furnace (GPF) is a three-zone resistance furnace which functionally duplicates one of the three furnace modules comprising the space shuttle flight furnace system (insert photograph). The electronic control of the MMSL furnace can independently control and monitor the temperature of each zone. Melting and cooling of materials in this furnace are currently being used to study macrosegregation phenomena in lead-tin alloys.

Electromagnetic Levitation Furnace

The electromagnetic levitation furnace (EML) is a 25-kW induction furnace used to perform containerless processing and undercooling experiments for conductive materials. The system can levitate and melt a metal or alloy sample in a vacuum or in an inert atmosphere. A flight version of the EML is currently available for performing materials experiments on the space shuttle.

Instrumented Drop Tube

Attached to the laboratory EML is a drop tube which provides 1 sec of low-gravity freefall. This system which allows the study of materials solidified in reduced gravity, is the only experimental system in the MMSL that provides a low-gravity environment. The specimens are tracked with infrared sensors as they fall down the 16-ft tube. Changes in specimen temperature are monitored by optical pyrometers positioned along the length of the tube.

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7. Freche, J.C.; and Waters, W.J.: Nickel Base Alloy. U.S. Patent No. 4,046,560, September 6, 1977.
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11. Bill, R.C.; and Ludwig, L.P.: Composite Seal for Turbomachinery. U.S. Patent No. 4,135,851, January 23, 1979.
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16. Stephens, J.R.; and Witzke, W.R.: High Toughness—High Strength Iron Alloy. U.S. Patent No. 4,214,902, July 29, 1980.
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19. Levine, S.R.; Miller, R.A.; and Hodge, P.E.: Corrosion Resistant Thermal Barrier Coating. U.S. Patent No. 4,255,495, March 10, 1981.
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IR–100 AWARDS

Each year "100 of the most significant new technical products of the year" are selected from industry, acadamia, and Government laboratories by Industrial Research Magazine. NASA Lewis has had many contributions chosen. The following are Lewis' materials research contributions that have received the IR–100 award.

Stable HC Bearing Materials—1966
Ductile Precipitate-Strengthened Tungsten Alloy—1967
Refractory Fiber Reinforced Superalloy—1968
Ferromagnetic Superalloy—1968
High-Purity Metal Powders—1971
Floating Zone Fiber Drawing Process—1972
Gas Lubrication Self-Acting Seals—1973
Oxidation-Resistant Self-Lubricating Bearing Materials for Use to Temperature from 25 to 900 °C—1974
High-Strength Nickel-Base Alloy, WAZ–16—1974
Advanced Ball Bearing Design for 3-Million DN Operation—1975
Ceramic Thermal Barrier Coating—1976
Second Generation PMR Polyimides—1977
Low Temperature Alloys (Low Chromium Stainless Steel, Tough—Strong Iron Alloy)—1978
Superalloy Strength Enhancement Fabrication Process—1978
High Efficiency Practical Magnetic Heat Pump—1978
Corrosion Resistant Thermal Barrier Coating—1979
NASVY/TRAC Multiroller Traction Drive—1979
MA 6000E Oxide Dispersion Strengthening Superalloy—1980